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56

MCR-67-18
(Issue 7)

JPL Contract 951709

STERILIZABLE LIQUID PROPULSION SYSTEM

Quarterly Report

October 1968

Abstract

The purpose of this program was to design, fabricate and make a demonstration firing of a fueled bipropellant liquid propulsion module that is capable of withstanding exposure to a sterilization environment. This seventh quarterly progress report covers effort under an extension to the basic contract. Work has been initiated to refurbish the module after which it will be loaded with propellants, exposed to additional sterilization and will again be fired. Design modifications have been implemented in both propellant tanks. A new equational diaphragm joint design and a modified trap design have been initiated.

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(Issue 7)

JPL Contract 951709

STERILIZABLE LIQUID PROPULSION SYSTEM

Seventh Quarterly Progress Report

October 1968

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FOREWORD

This document is the seventh issue of the Quarterly Progress Report and is submitted in accordance with Article 1(a)(1)(v)(E) and 2(b)(5) of JPL Contract 951709.

CONTENTS

I. INTRODUCTION

This is the seventh quarterly progress report submitted in accordance with JPL contract 951709. This report covers the period from 1 July through 30 September 1968.

The program involved the design assembly and exposure of the fueled bipropellant liquid propulsion system to the ethylene oxide (ETO) and heat sterilization environments specified by JPL specification VOL50503 ETS. After exposure, the system was successfully fired for 280 seconds. The program was supported by a materials compatibility test program and component verification program wherein suitability of all selected components was demonstrated prior to the system assembly.

Under an extension to the contract the system will be refurbished, submitted to additional heat sterilization and fired again. Design changes will be made to the oxidizer tank and expulsion diaphragm during the rebuild period. In addition the fuel tank screen trap will be modified to eliminate two-phase flow from the tank when expelling in a negative 1-g regime.

During the report period improvised flow tests of the screen trap were performed in a Plexiglass Hemisphere that pin pointed the cause of the two-phase flow. Corrective action was determined, demonstrated and implemented.

II. CONCLUSIONS

As a result of the work performed during this period, it was concluded the two-phase flow from the fuel tank in a negative 1-g regime was caused by leakage through the flat plate riveted joint in the trap that made the transition from steel to titanium so the trap could be welded into the titanium tank.

Further it was concluded the Marquardt R4-D engine suffered no significant performance degradation as a result of the sterilization program.

III. RECOMMENDATIONS

It is recommended that a new program be implemented to develop compatible screen material with the propellant tanks. As a result of the repeated problems encountered in the attachment of the screen trap containing steel screens to a dissimilar tank material, development of etched titanium foil or titanium screens should be initiated.

IV. GENERAL REPORT

A. SCREEN TRAP TESTING

The fuel tank from the module assembly was removed and sectioned for evaluation. The fuel trap from the module showed a leak check value of 0.5" H_2O as compared to 1.5" H_2O of the trap from the component tank. The leakage path was through the rivet holes on the lower closure plate of the trap which was the same as that experienced on the component tank. This type of leakage degradation was a basic result of the sterilization program. All screens, filters and rivet closures degraded at least 30%.

Analysis of the propellant flow path under the trap of the component tank revealed that due to shop tolerances a pressure drop in excess of 1.5" H_2O could be expected and a break down of the screen permitting two-phase flow could result. Since both trap units showed a severe degradation of the riveted closure plate, a new design that eliminated the requirement for a lower closure with a cherry rivet fastening technique was made. Figure 1 shows the new configuration of the improved screen trap assembly which with the welded closure is insensitive to the flow pressure drop shown in the next paragraph.

Machining of the lower shelf to reweld the trap into the fuel tank will be controlled to yield an average flow path clearance of 0.082 inches which will result in an equivalent orifice diameter of 0.971 inches. This will produce a calculated fuel pressure drop of 0.127" H_2O.

Because of the better condition of the screens, the screen trap assembly from the module tank was selected for modification and reinstallation in the module tank since the closure patch with attendant lower leak check level was eliminated.

Further testing of a screen trap assembly was accomplished during the report period. The purpose of the testing was to visually determine the flow characteristics of the trap and the adequacy of the propellant loading technique employed during the program. A crude test fixture was devised utilizing a clear Plexiglass hemisphere from another program. The trap was held in a hemisphere 10 inches in diameter with sealing putty.

Several tests clearly showed the smallest bubble was resident inside the trap when filled in the inverted position. This was the

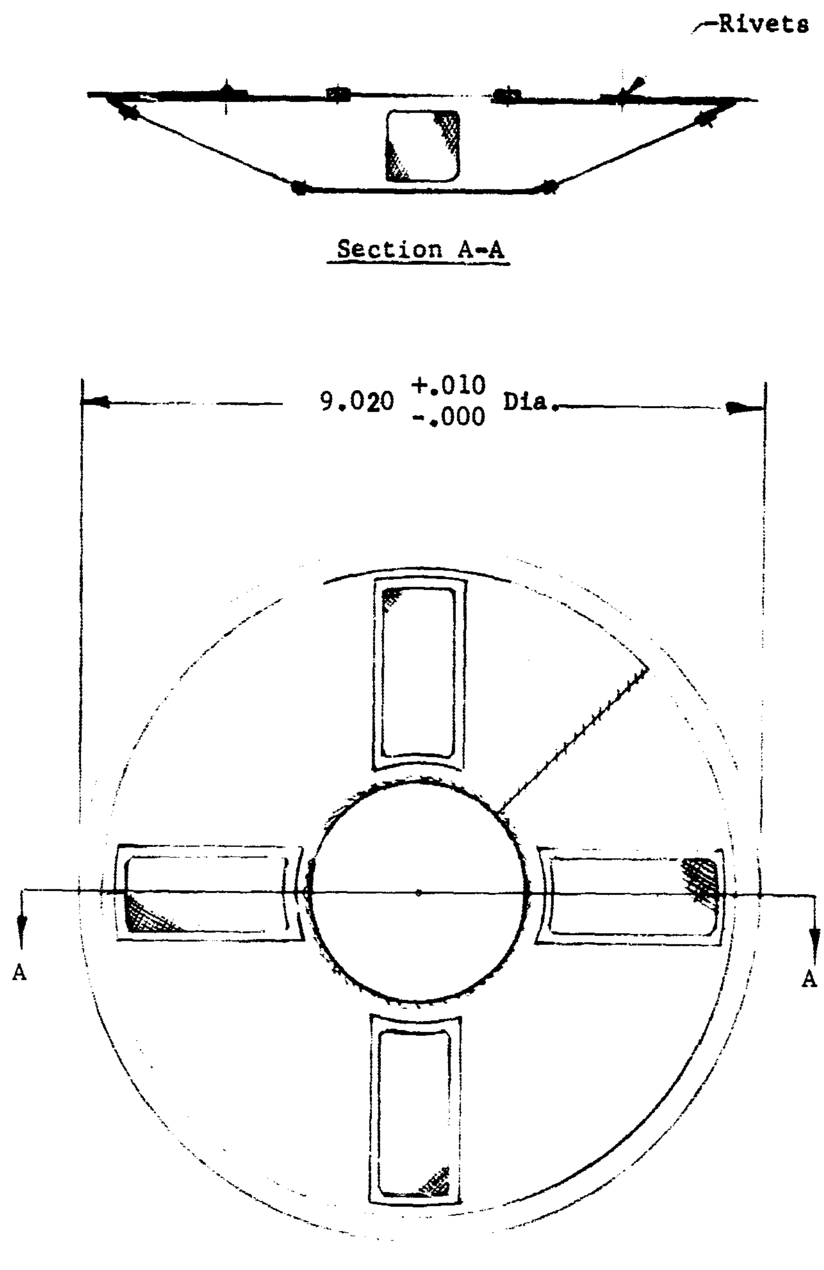


Figure 1 Improved Screen Assembly

technique employed in loading the module. During the outflow tests, bubbles were observed flowing through the attachment area between the titanium ring and the steel body. This joint is formed by a ring of rivets which were leak tight through the joint; however, the leak path was between the surfaces shown by the arrow in figure 2.

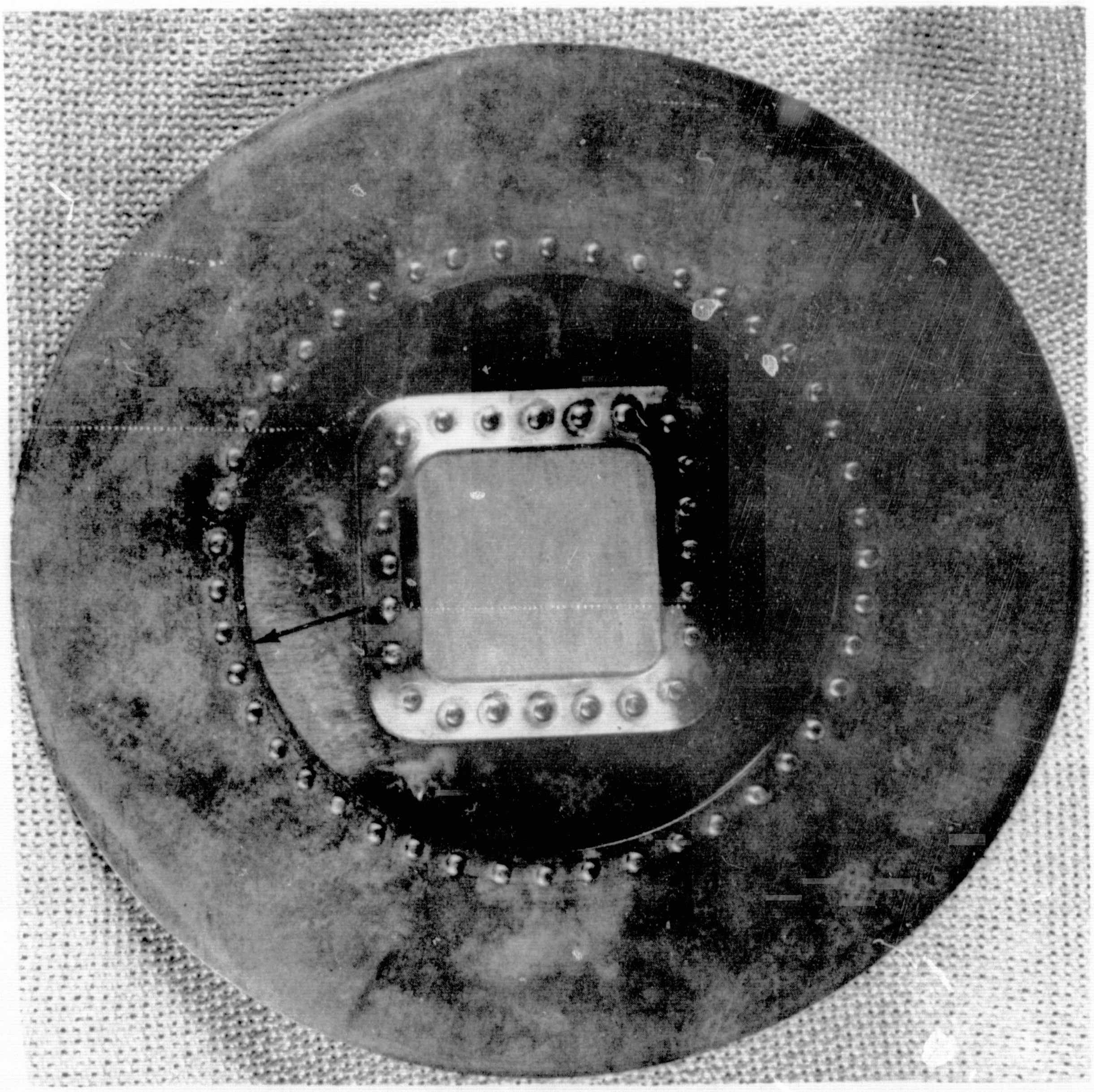


Fig. 2: Typical Trap Assembly Showing Leak Path

This series of tests clearly showed bubbles emerging from the ring area resulting in a well established two-phase flow. Sealing the joint with putty resulted in the desired single-phase liquid flow.

On this basis and on the assumption that this would occur in the final assembled unit to be installed in the module, a search was initiated to find a compatible method of sealing the titanium to stainless steel surface at the rivet diameter. First, the metallic type of sealants were considered such as soft solders and brazes. The low temperature solders and brazes, up to 1200°F, were discarded because the fluxes necessary to wet the titanium might cause the fuel to decompose. One high temperature braze of gold-nickel could be accomplished, but this would require a brazing temperature of approximately 1800°F. This high a temperature might cause the rivets to loosen or the screen to degrade, a risk that was not attractive therefore the metallic sealants were discarded from further consideration, and an attention was focused on the non-metallic sealants. The non metallic sealants had to be tested to prove sealing ability and compatibility with the fuel (MMH) at the sterilizing temperature of 135°C.

A series of candidate sealants were tested for qualification. The tests were:

1) Initial tests at room temperature in monomethyl hydrazine

2) Sample joints were made to test for leak tightness before and after fuel exposure.

3) Exposure of the sealed joints in fuel at 135°C for 114 hours.

Table 1 presents the candidate materials and the results of the testing.

During the initial testing certain results were clouded because of inadequate cleaning procedures. All subsequent test specimens were cleaned as follows:

1) Tri-Sodium phosphate bath (20 minutes)

2) Water rinse

3) Immerse in Turco de-oxidizing solution (15 minutes)

4) Water Rinse

5) Quick dip in nitric acid + HF

6) Rinse in demineralized water

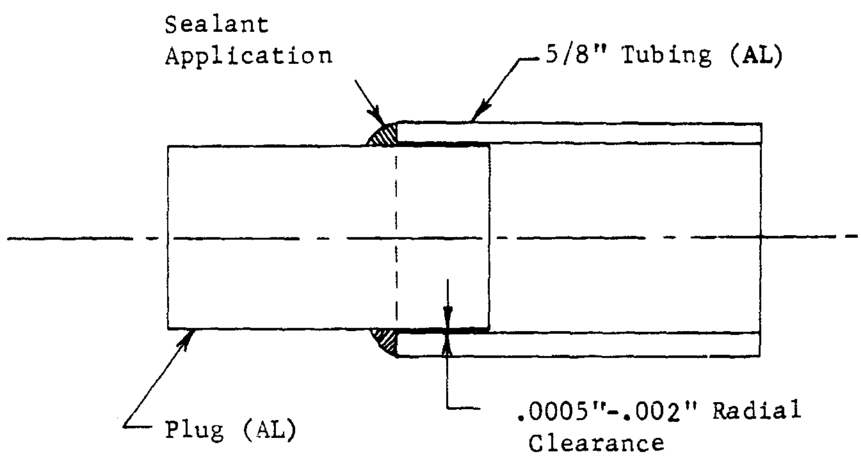
7) Dry

The joints made up for the final immersion in hot MMH were fabricated as shown in Fig. 3. All the sealants were set aside for 24 hours to provide a room temperature cure. All the joints except the DC-11 were leak tested to 3 psi. The joints were then placed in bombs, loaded to a 30% ullage with MMH, sealed and placed in a bath at 135°C. Pressures rose no higher than 88 psia which compares favorably to the normal vapor pressure of 63 psia. Pressures

TABLE I

RESULTS OF NON-METALLIC SEAL EXPOSURE IN MONOMETHYL HYDRAZINE

Material	Room Temp. Tests 72 hour	135°C 114 hours
1. Locktile, grade H	OK	Lost Adhesion
2. Silicone Sealant MMSK-138	Lost Adhesiveness Eliminated from Further Testing	
3. Clear Seal - a GE Flexible Adhesive of Translucent Silicone Rubber	OK	Maintained Seal: Excess Bead Lost All Adhesion
4. Elasto-Coat #1-2020 a Chemical Milling Mask Manufactured by Organo-Cerams Inc.	Lost Adhesiveness Eliminated from Further Testing	
5. Water Glass, Sodium Meta-Silicate: Na_2O x SiO_2 (x = 2 - 5)	OK	Good Hard Bead no Leakage
6. Dow Corning DC-11 Grease	OK	Dissolved - Seal Destroyed



TEST JOINT

Fig. 3

were checked at 24, 48 and finally at the conclusion of the test at 114 hours. No significant change occurred indicating that none of the materials caused decomposition of the fuel.

To assure ourselves the available trap assembly from the component tank could be properly treated and tested with the water glass, a demonstration was performed. The questionable joint was cleaned with an alkaline solution and water rinsed. Upon air drying methyl ethyl ketone was swabbed around the joint and dried. The water glass was then applied and allowed to dry for 48 hours, see Fig. 4. The unit was then assembled into the hemisphere and water flow tests proved conclusively the leak between the riveted plates had been stopped as indicated by single-phase liquid flow. These procedures will be employed for the trap installed in the modified tank prior to the final closure weld.

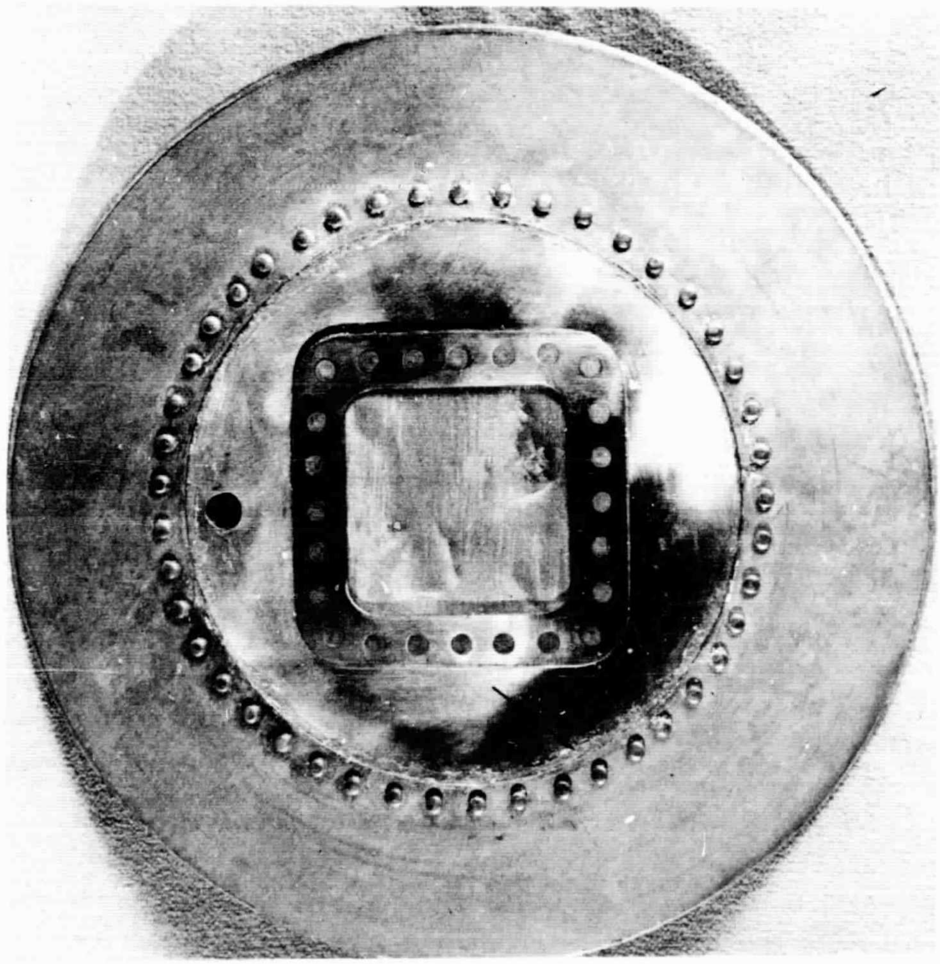


Fig. 4: Fuel Trap Assembly Showing Water-Glass Seal After Test

B. SCREEN TRAP FABRICATION

The screen trap selected for modification and installation in the rebuilt module oxidizer tank was welded in a small evacuated chamber. The larger Martin Marietta weld chamber was out of commission for modification at the time, and the single trap unit fabrication did not warrant reprogramming of that activity. The completed unit was submitted for passivation in monomethyl hydrazine (MMH) on July 31. Repeated attempts to passivate the unit in the fuel were unsuccessful as manifested by continued evolution of gas bubbles signifying decomposition of the MMH. The unit was then immersed in hydrazine in hopes that the hydrazine would reduce the surface catalytic reaction by reducing the surface oxides. This procedure was unsuccessful as noted by continued formation of gas bubbles though at a somewhat slower rate.

A sophisticated cleaning process was devised to clean the trap unit without destroying the screen cloth. The process included the steps outlined below:

- Mask the screens
- Clean outside surface with alkaline solution such as Cee Bee Industrial Cleaner
- Clean outside surface with HNO_3 + HF agent. The cleaning agent was made up of 40% HNO_3/60%H_2O + 8% HF. Contact with the screens was to be avoided.
- Immerse entire unit in a 40% HNO_3 solution.
- Rinse in demineralized water.

When the process was implemented, the alkaline cleaner made the surface clean. Application of the HNO_3 + HF foamed considerably. The HNO_3 + HF was repeated at least 4 times after which all foaming stopped. The unit was then placed in a bath of hot N_2H_4 at 202°F. (local water boiling temperature) for 24 hours. Following the successful verification of passivation, the unit was cleaned in demineralized water followed by an acetone rinse and shipped to the vendor for installation in the tank on 9 August, 1968. The completed trap assembly is shown in Fig. 5 and 6.

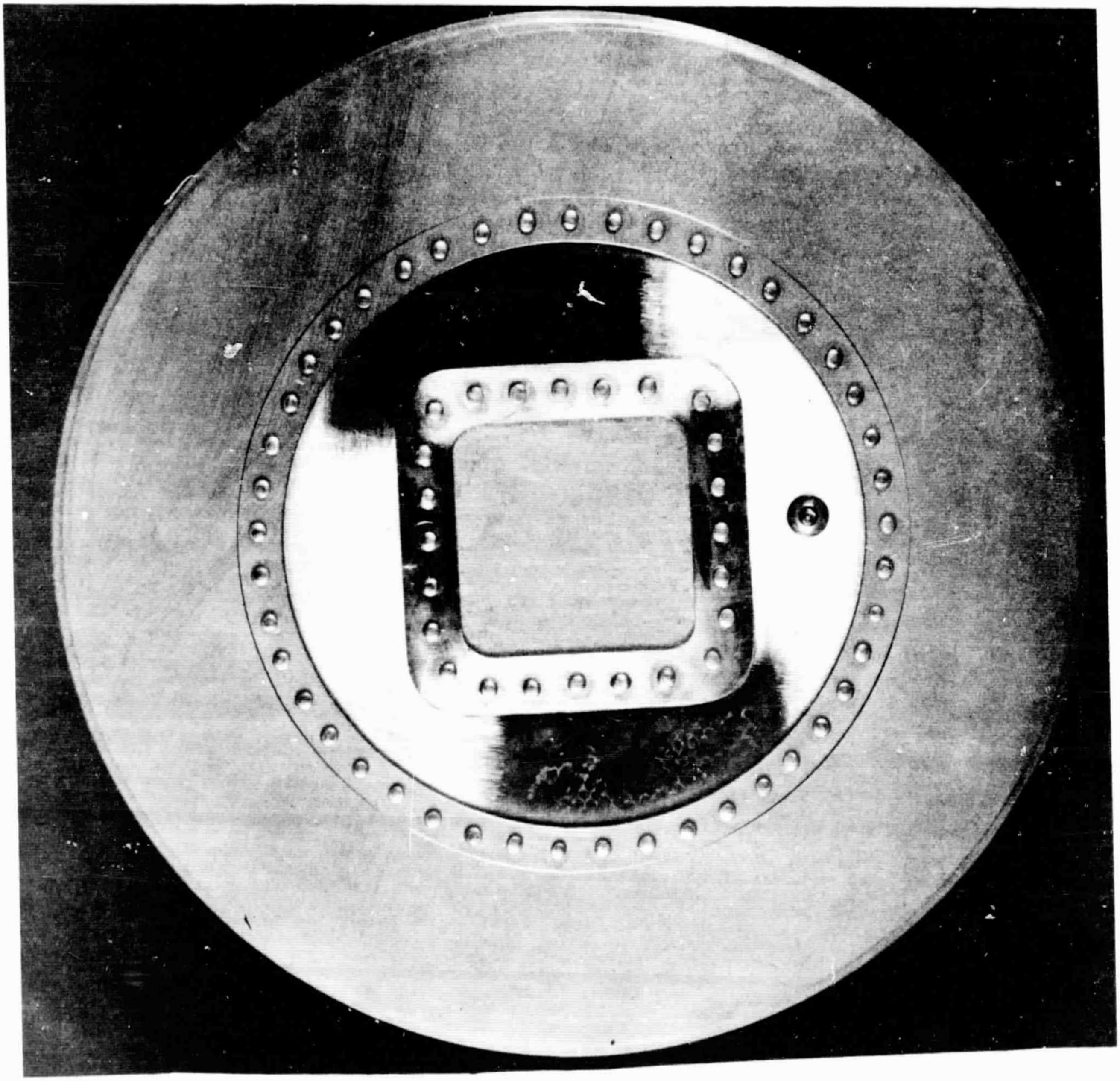


Fig. 5: Modified Trap Assembly Top View

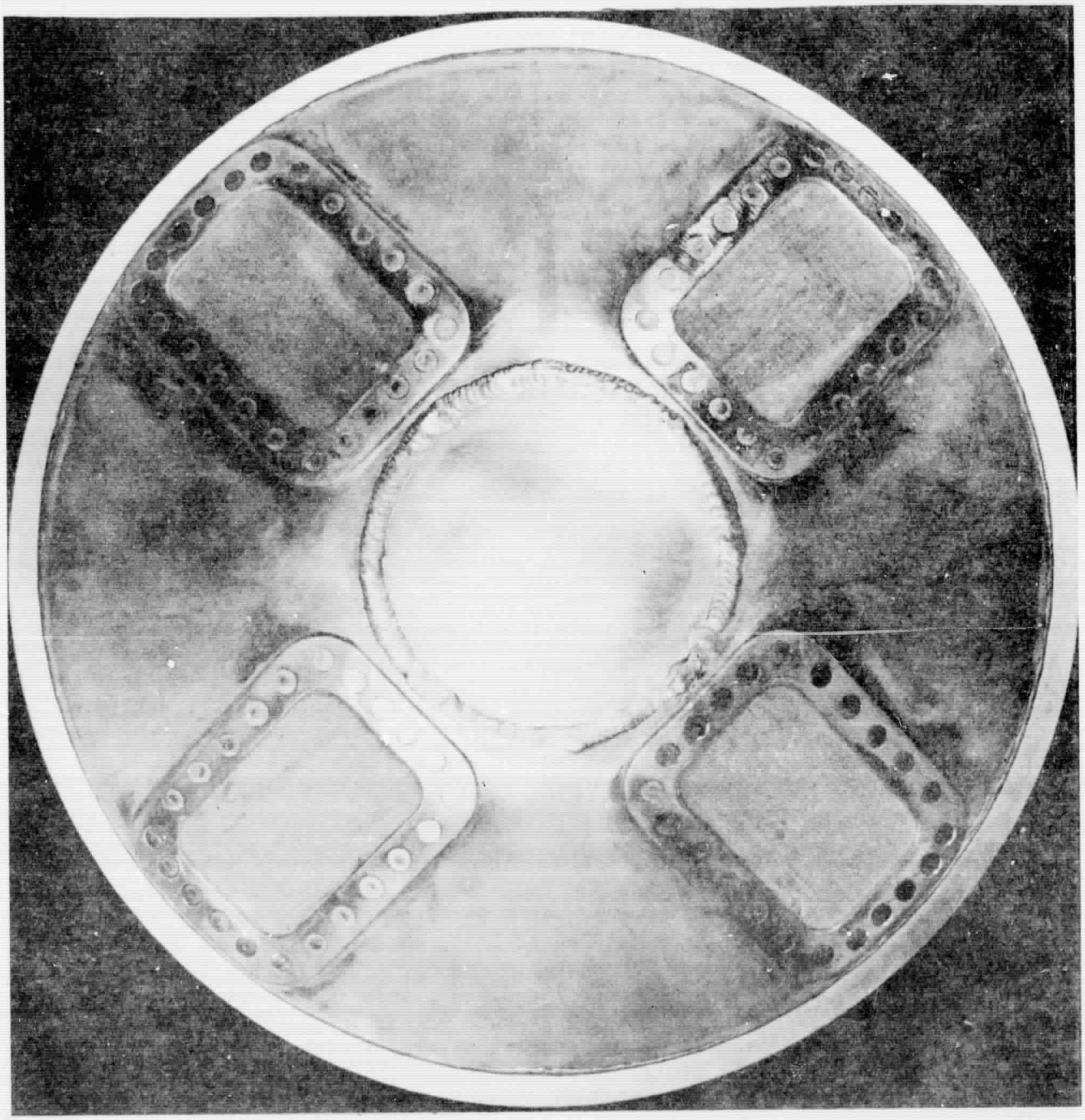


Fig. 6: Modified Trap Assembly Bottom View

C. SUBCONTRACT ACTIVITY

1) Marquardt Corporation

The Marquardt R4-D engine, part number X-229663 S/N 0001, was returned to the Marquardt Corporation in July, 1968, for inspection and water flow check. To preserve the conditions of the engine for valid continuation of the sterilization program, it was not disassembled. Table 2 presents representative engine characteristics before and after the sterilization and firing test.

TABLE 2

Engine Characteristics

	Pretest	Post Test	Change
Oxid. Flow H_2O/PPH	552	555	+ .5
Oxid Response ms	8.3	8.4	+1.2
Oxid Leakage SCC/HR	0	0	0
Fuel Flow PPH-H_2O	438	436	- .5
Fuel Response ms	6.4	6.4	0
Fuel Leakage SCC/HR	0.50	0	-100

From the results shown above, the engine suffered insignificant degradation as a result of the first sterilization program. On this basis, no adjustments will be made to the flow control orifices. The engine was returned to Martin Marietta Corporation and reinstalled in the module.

2) Sterer Engineering and Manufacturing Company.

The solenoid valve incorporated in the module was Sterer P/N 35580, S/N 2. After the sterilization exposure and firing, the 500 VDC resistance from coil to case was less than 1 megohm. As a result, the #27 Formvar wire which does not hold up at 135°C was replaced. The new coil winding incorporates SML magnet wire.. The insulation of the bobbin spool and end plates is Micomat wet wound with DC-997 high temperature varnish. All solder attachments, sleeving, and insulation are of high temperature materials. The finish coil is wrapped with a teflon tape type P-421. The assembled unit was then baked at 400°F for 1 hour. The valve was acceptance tested and conformed to all requirements. The valve has been returned to the Martin Marietta Corporation for reinstallation in the module.

3) Dilectrix Corporation

Fabrication of 2 units of the Teflon diaphragm was completed late in the report period. Fig. 7 shows the general configuration of the diaphragm. During the fabrication of the diaphragms, some difficulty was encountered in obtaining a complete and uniform bond between the TFE/FEP diaphragm and the ring flange covered with codispersion Teflon. The bonding process was repeated 3 times on unit #1 in an attempt to obtain a complete bond. After the bonding, some small pockets of unbonded Teflon were evident even though the leakage test was successfully completed. The unit was accepted on the basis of the successful leak check in the absence of a well defined bond criteria. The unit will be installed in the component tank as is to gather additional experience and data to develop the bonding criteria. Inspection of the bonding area indicated that the Teflon was beginning to flow and thus form sharp corners around the ring flange. A dimensional check revealed that the critical dimension is in excess of the drawing 0.003." The dimension is critical in that if it were under size it would affect the grip of the tank halves shown by fig. 8. Since it was slightly in excess of the required valve, it was decided to not rework the diaphragm. The next report will show photographs of the condition of the bonded flange area. At the close of the report period, the units were awaiting shipment to the Martin Marietta Corporation Denver Division.

4) Pressure Systems Corporation

Two oxidizer tanks and one fuel tank were returned to PSI for rework. The fuel tank halves are to be rewelded to the original configuration. Delivery of the fuel tank is expected during October 1968.

The oxidizer tanks are to be modified as shown in Fig. 8. The modifications to the tanks consist of mounting the diaphragm on the tank equator so that the diaphragm is completely retained by the tank wall in either the normal or expelled condition thereby precluding an inadvertent rupture due to over expulsion. Because of the lead times involved in the procurement of the forging blanks by PSI, the delivery of the oxidizer tanks are not expected until late December 1968.

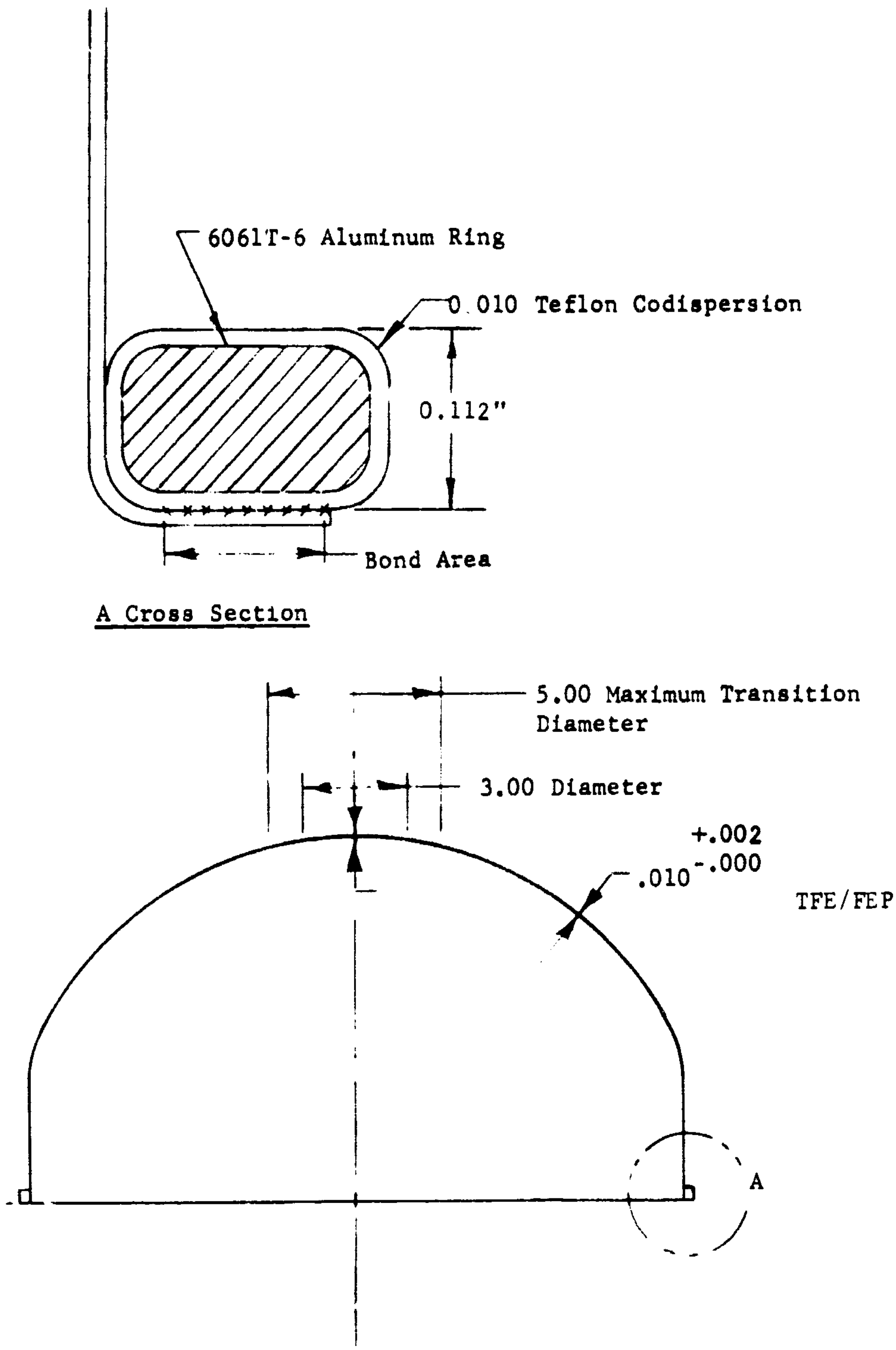


Figure 7. Oxidizer Tank Diaphragm Design

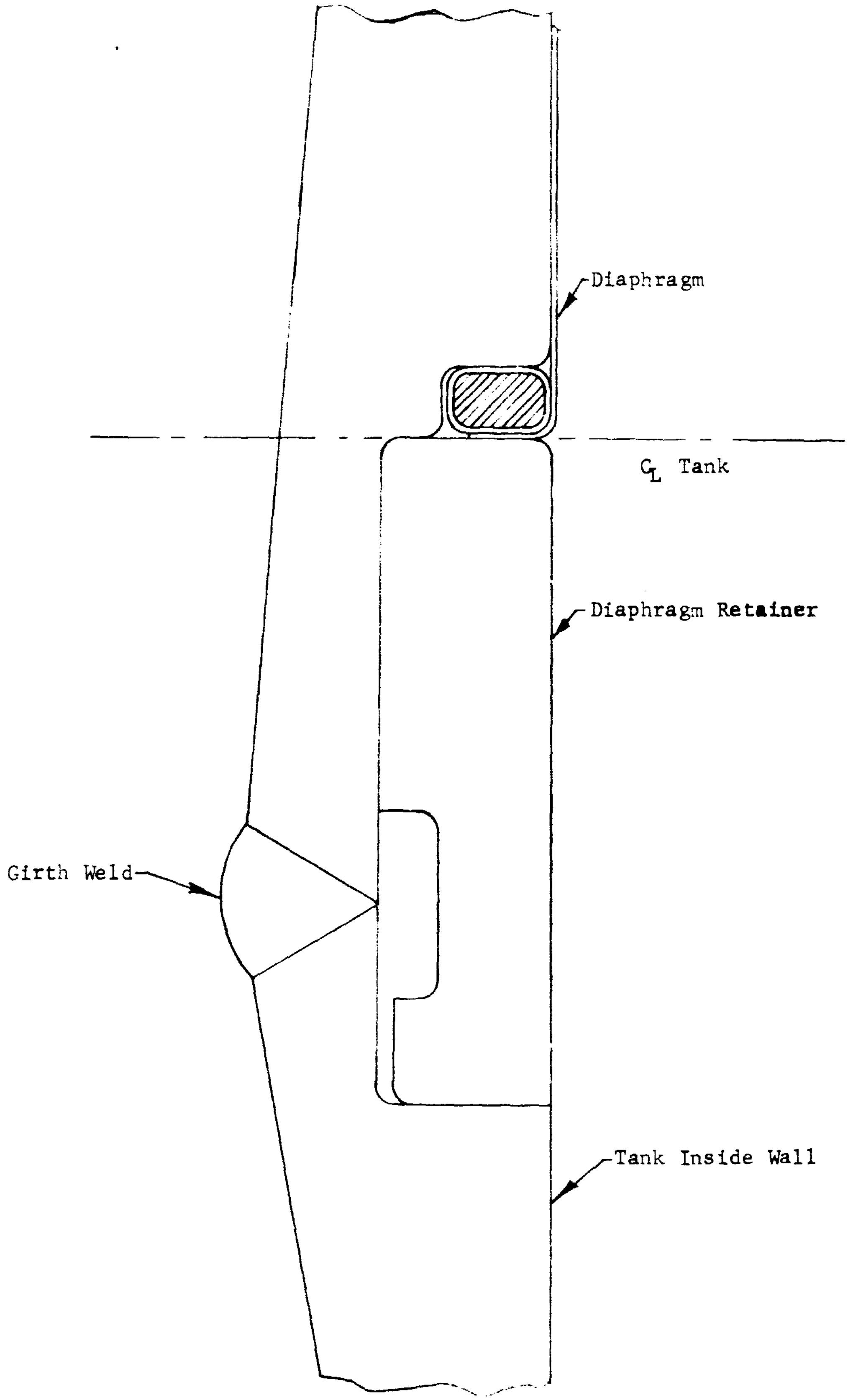


Figure 8. Oxidizer Tank Diaphragm Retainer Joint Design

D. PROBLEM AREAS

During the course of the program, the tank end fittings that were removed from the tanks so the tanks could be mounted in a lathe for sectioning have been misplaced. This necessitates remachining two fittings from titanium that attach the ordnance valve to the propellant tank. A welded part of the fitting assembly is a transition tube that accommodates the tank fill or vent line as the case may be. The tube makes the transition from titanium 6 Al- V to 6061-T6 Aluminum. New tubes have been put on order. The remanufacture of these items will impose no schedule slippages since sufficient lead time remains.

The condition of one unit of the Teflon diaphragms presents a problem in bonding criteria of the diaphragm to the ring flange. This unit will be installed in the component tank unit for further evaluation.